

(i) providing said aqueous solvent in an amount resulting in complete hydrolysis and providing said acid in an amount maintaining a hydrolyzed precursor and avoiding gelation or precipitation; and

(ii) providing said surfactant and said silica precursor in a mole ratio that is above a lower mole ratio that produces a non-porous silica phase and below an upper mole ratio that produces a lamellar phase.

2. The method as recited in claim 1, wherein said lower mole ratio is about 0.05.
3. The method as recited in claim 1, wherein said upper mole ratio is about 0.3.
4. The method as recited in claim 1, wherein said acid is added in an amount resulting in a pH of said silica precursor solution of from about 1 to about 4.
5. The method as recited in claim 4, wherein said pH is about 2.
6. The method as recited in claim 1, wherein the step of forming includes diluting with an alcohol.
7. The method as recited in claim 6, wherein said alcohol is ethanol.
8. The method as recited in claim 1, wherein said aqueous solvent, said acid, and said surfactant are premixed before combining with said silica precursor.
9. The method as recited in claim 1, wherein said mesoporous material is in a geometric form selected from the group consisting of fiber, powder, and film.
10. The method as recited in claim 1, wherein said forming is spin-casting.
11. The method as recited in claim 1, wherein said forming is spraying.

12. The method as recited in claim 1, further comprising adding a pre-polymer or a polymer to said silica precursor solution making a pituitous mixture.
13. The method as recited in claim 1, wherein said forming is drawing.
14. The method as recited in claim 1, wherein said forming is squeegeeing.
15. The method as recited in claim 1, further comprising the step of adding a metal compound to the silica precursor solution.
16. The method as recited in claim 15, wherein said metal compound is selected from the group consisting of metal halide, metal nitrate, and combinations thereof.
17. The method as recited in claim 16, wherein said metal halide is a metal chloride.
18. The method as recited in claim 16, wherein said metal is selected from the group of aluminum, iron and combinations thereof.
19. The method as recited in claim 1, wherein said silica precursor is an alkoxide silica precursor or a tetrachlorosilane.
20. The method as recited in claim 1, wherein said aqueous solvent amount is characterized by a ratio of said aqueous solvent to said silica precursor of about 7.
21. The method as recited in claim 1, wherein said acid amount is characterized by a ratio of said acid to said silica precursor of about 0.1.
22. The method as recited in claim 1, further comprising adding a swelling agent to the silica precursor solution.

23. The method as recited in claim 22, wherein said swelling agent is 1,3,5-trimethylbenzene.

24. The method as recited in claim 1, further comprising the step of calcining the mesoporous material.

25. A method of making a mesoporous silica film, comprising the steps of

(a) combining a silica precursor with an aqueous solvent, an acid and a surfactant having an ammonium cation into a silica precursor solution,

(b) templating the silica precursor with the surfactant and obtaining the mesoporous material from the templated silica precursor,

(c) forming said silica precursor into a preform; and

(d) rapidly evaporating said aqueous solvent from said preform for obtaining the mesoporous material, wherein the improvement comprises:

(i) said silica precursor is tetraethoxysilane;

(ii) providing said aqueous solvent in a superstoichiometric amount and providing said acid in an amount maintaining a hydrolyzed precursor and avoiding gelation or precipitation;

(iii) providing said surfactant and said silica precursor in a mole ratio that is above a lower mole ratio that produces a non-porous silica phase and below an upper mole ratio that produces a lamellar phase; and

(iv) said forming includes diluting with an alcohol.

26. The method as recited in claim 26, further comprising adding a pre-polymer or a polymer to said silica precursor solution making a pituitous mixture.

27. The method as recited in claim 26, wherein said rapidly evaporating is by spin-casting.

28.-39. Cancel.

40. (Amended) A method of making a mesoporous film on a substrate, the method comprising the steps of:

- 21
- (a) combining a silica precursor with an aqueous solvent, an acid catalyst and an ammonium cationic surfactant into a precursor solution;
 - (b) dispensing said precursor solution onto the substrate;
 - (c) forming a film by evaporation of the solvent in less than 5 minutes; and
 - (d) heating the film on the substrate to a temperature sufficient to decompose the surfactant, thereby producing a mesoporous film on the substrate.

41. The method of claim 40 wherein the precursor solution is a silica precursor solution and wherein the surfactant and the silica precursor solution are in a mole ratio that is above a lower mole ratio that produces a non-mesoporous silica phase and below an upper mole ratio that produces a lamellar phase.

58. (Thrice amended) A process to form mesostructured films, comprising:

- 22
- (a) preparing a precursor sol containing a soluble source of silica, an aqueous solvent, an ammonium cationic surfactant and an acid catalyst; and
 - (b) depositing the precursor sol on a substrate wherein evaporation of solvent and water in less than 5 minutes causes the formation of said mesostructured films on the substrate surface.

59. (Twice amended) The process of claim 58 wherein the aqueous solvent and the catalyst are provided in amounts that maintain a hydrolyzed precursor sol while avoiding gelation or precipitation.

60. (Twice amended) The process of claim 58 wherein the soluble source of silica is a silica precursor alkoxide or tetrachlorosilane and wherein the surfactant and the soluble source of silica are in a mole ratio that is above a lower mole ratio that produces a non-porous silica phase and below an upper mole ratio that produces a lamellar phase.

23
June 29, 2001

69. (Twice amended) The process of claim 58, wherein the ammonium cationic surfactant further includes bromide or chloride or their combination.

new matter new matter

RESPONSE TO
OFFICE ACTION

PAGE 5 of 22

DO. NO. 1941-70

70. (Twice amended) The process of claim 58, further comprising the step of calcining said film at 450°C.

71. The process of claim 58, wherein the precursor sol is deposited on a substrate by spin coating.

78. (Amended) The process of claim 58, wherein said soluble source of silica is an alkoxide silica precursor or tetrachlorosilane.

79. (Amended) A process to form a mesoporous structure, comprising:

- (a) preparing a precursor sol containing a soluble source of silica, an alcohol and water solvent, an ammonium cationic surfactant, and an acid catalyst, wherein said solvent is provided in an amount resulting in complete hydrolysis and said acid catalyst is in an amount to maintain a hydrolyzed precursor and to avoid gelation or precipitation in said precursor sol;
- (b) forming the precursor sol into a preform;
- (c) evaporating said solvent from the preform at a rate that forms a mesostructured material; and
- (d) calcining the mesostructured material to form a mesoporous structure.

80. The process of claim 79, wherein said precursor sol contains alcohol which is a byproduct of hydrolysis, and said mesoporous structure is a film.

81. The process of claim 79, wherein said preform is a droplet, said alcohol is a byproduct of hydrolysis, and said sol is spray dried to form a powder.

82. The process of claim 79, wherein said drying is preformed in less than 5 minutes.

83. (Amended) The process of claim 79, wherein said precursor sol contains dilutant alcohol, and wherein the mesoporous structure is a film.

RESPONSE TO
OFFICE ACTION

PAGE 6 of 22

DO. NO. 1941-70

84. A process to form a mesoporous structure, comprising:

- (a) preparing a precursor sol containing a soluble source of silica, an alcohol and water solvent, an ammonium cationic surfactant, and an acid catalyst, wherein said solvent is provided in an amount resulting in complete hydrolysis and said acid is in amount to maintain a hydrolyzed precursor and to avoid gelation or precipitation in said precursor sol;
- (b) forming the precursor sol into a preform;
- (c) evaporating said solvent from the preform at a rate that forms a mesostructured material, wherein said mesostructured material contains surfactant; and
- (d) calcining the mesostructured material to form a mesoporous structure.

D6 ~~85.~~ (Amended) A process to form a mesostructure, comprising:

- (a) preparing a precursor sol containing a soluble source of silica, water and alcohol solvent, an ammonium cationic surfactant and a catalyst, and
 - (b) evaporating said solvent in less than 5 minutes to cause the formation of a mesostructure, wherein said mesostructure contains surfactant.
- limited to ACID catalyst*

86. The process of claim 79, wherein the said precursor sol contains alcohol which is a byproduct of hydrolysis, and wherein said mesostructure is a film.

87. The process of claim 79, wherein said preform is a droplet, wherein said alcohol is a byproduct of hydrolysis, and wherein said precursor sol is spray dried.

88. The process of claim 79, wherein said evaporating is performed in less than 5 minutes.

89. Cancel ✓

D7 90. (Amended) The process of claim 79, wherein said soluble source of silica includes a silica alkoxide precursor or tetrachlorosilane.

RESPONSE TO
OFFICE ACTION

PAGE 7 of 22

DO. NO. 1941-70

91. A process to form a mesostructure, comprising:

- OK support from original claim 6 —*
- (a) preparing a precursor sol containing a soluble source of silica, a water and alcohol solvent, an ammonium cationic surfactant and an acid catalyst, and
- (b) evaporating said solvent in less than 5 minutes to cause the formation of a mesostructure.

D8

92. (Amended) The process of claim 91, wherein said solvent is evaporated in less than 1 minute.

93. (Amended) The process of claim 91, wherein said solvent is evaporated in less than 10 seconds.

new matter

94. The process of claim 91, wherein the said precursor sol contains both dilutant alcohol and alcohol which is a byproduct of hydrolysis, and wherein said mesostructure is a film.

limited to ... (or the other spec ...)

95. The process of claim 91, wherein said preform is a droplet, said alcohol is a byproduct of hydrolysis, and said sol is spray dried.

96. Cancel.

97. Cancel.

D9/103

98. (Amended) The process of claim 91, wherein the ammonium cationic surfactant further includes bromide or chloride or their combination.

new matter

99.-108. Cancel.

D10

109. (Amended) The process of claim 40, wherein the film exhibits an index of refraction between 1.16 and that of silica.

RESPONSE TO
OFFICE ACTION

PAGE 8 of 22

DO. NO. 1941-70

110. (Amended) The process of claim 58, wherein the films exhibit an index of refraction between 1.16 and that of silica.

111. (Amended) The process of claim 79, wherein the mesoporous structure is a film and wherein the film exhibits an index of refraction of between 1.16 and that of silica.

112. (Amended) The process of claim 85, wherein the mesostructure is a film, and wherein the film exhibits an index of refraction of between 1.16 and that of silica.

113. (Amended) The process of claim 86, wherein the film exhibits an index of refraction of between 1.16 and that of silica.

114. (Amended) The process of claim 92, wherein the mesostructure is a film, and wherein the film exhibits an index of refraction of between 1.16 and that of silica.

115. (Amended) The process of claim 99, wherein the films exhibit an index of refraction of between 1.16 and that of silica.

116. (New) An improved method of forming templated mesoporous material on a substrate from a silica precursor solution containing an alkoxide silica precursor, and ammonium cationic surfactant and a solvent, while avoiding gelation, precipitation and non-porous or lamellar structures, wherein the improvement comprises the steps of:

- (a) preparing said silica precursor solution using a solvent;
- (b) dispensing a layer of said precursor solution on said substrate;
- (c) thinning said layer by spin casting; and
- (d) forming templated mesoporous material on said substrate by evaporation of the solvent in less than 5 minutes.

117. (New) An improved method of forming templated mesoporous material from a silica precursor solution containing an alkoxide silica precursor, an ammonium cationic surfactant and a solvent, while avoiding gelation or precipitation or non-porous or lamellar structures, wherein the improvement comprises the steps of: *new water*

- new matter
- (a) preparing said silica precursor solution using a solvent;
- (b) spin casting, drawing, spraying or squeegeeing said silica precursor solution; and
- (c) evaporating the solvent in less than 5 minutes to form templated mesoporous material.

118. (New) An improved method of forming templated mesoporous material on a substrate from a silica precursor solution containing an alkoxide silica precursor, an amount of surfactant ^{new matter} great enough to avoid a non-porous film but not enough to produce a lamellar structure, and a solvent, while avoiding gelation or precipitation, wherein the improvement comprises the steps of:

- (a) preparing said silica precursor solution using a solvent;
- (b) dispensing a layer of said precursor solution on said substrate;
- (c) thinning said layer by spin casting; and
- (d) forming templated mesoporous material on said substrate by evaporation of the solvent in less than 5 minutes.

119. (New) An improved method of forming templated mesoporous material from a silica precursor solution containing an alkoxide silica precursor, a surfactant and a solvent, while avoiding gelation or precipitation and non-porous or lamellar structures, wherein the improvement comprises the steps of:

- (a) preparing said silica precursor solution using a solvent;
- (b) spin casting, drawing, spraying or squeegeeing said silica precursor solution; and
- (c) evaporating the solvent in less than 5 minutes to form templated mesoporous material.
- 206K Chms 10-14*

120. (New) An improved method of forming templated mesoporous material on a substrate from a silica precursor solution containing an alkoxide silica precursor, and a solvent, while avoiding gelation or precipitation ^{OK} of non-porous or lamellar structures, wherein the improvement comprises the steps of:

- new matter*
- (a) preparing said silica precursor solution using a solvent;
 - (b) dispensing a layer of said precursor solution on said substrate;
 - (c) thinning said layer by spin casting; and
 - (d) forming templated mesoporous material on said substrate by evaporation of the solvent in less than 5 minutes.
- new matter* → *no surfactant!*
? not supported!
I spec would appear to require ammonia based surfactant

all cut

121. (New) An improved method of forming templated mesoporous material from a silica precursor solution containing an alkoxide silica precursor, and a solvent, while avoiding gelation or precipitation and non-porous or lamellar structures, wherein the improvement comprises the steps of:

- (a) preparing said silica precursor solution using a solvent;
 - (b) spin casting, drawing, spraying or squeegeeing said silica precursor solution; and
 - (c) evaporating the solvent in less than 5 minutes to form templated mesoporous material.
- agrees*
new matter → *no surfactant*
new matter